IS 6213 (Part 12) : 2013

(Reaffirmed 2018)

भारतीय मानक

लुगदी के लिए परीक्षण पद्धति

भाग 12 कैल्शियम सामग्री ज्ञात करना

(पहला पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PULP

PART 12 DETERMINATION OF CALCIUM CONTENT

(First Revision)

ICS 676.1: 676.014.2 : [546.41]

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Paper and Its Products Sectional Committee, CHD 15

FOREWORD

This Indian Standard (Part 12) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Paper and Its Products Sectional Committee has been approved by the Chemical Division Council.

Excessive amounts of calcium salts in pulp have an adverse effect on the quality of paper specially with regard to its sizing characteristics. It is, therefore, essential to know the calcium content in the washed bleached pulp before feeding it to the paper making machine.

This standard (Part 12) which was prepared mainly based on ISO/R 777 : 1968 'Pulps — Determination of calcium content' first published in 1975. As this version was very old, decision has been taken for its revision to update the standard. It is for information that International Organization for Standization (ISO) has published a standard ISO 12830 : 2011 'Paper, board and pulp — Determination of acid-soluble magnesium, calcium, manganese, iron, copper, sodium and potassium'. As this ISO method is for paper, board and pulp and IS 6213 (Part 12) prescribes method of test for pulp only, the Committee also decided to adopt ISO 12830 : 2011 'Paper, board and pulp — Determination of acid soluble magnesium, calcium, manganese, iron, copper, sodium and potassium' as dual number standard in future.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PULP

PART 12 DETERMINATION OF CALCIUM CONTENT

(First Revision)

1 SCOPE

This standard (Part 12) prescribes the method for the determination of calcium content of pulp.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6213 (Part 7) : 1971	Methods of test for pulp: Part 7 Ash content in pulp

3 PRINCIPLE OF THE METHOD

The pulp is ashed and the ash is dissolved in nitric acid. Calcium is determined volumetrically with a solution of the disodium salt of ethylenediaminetetraacetic acid at a *p*H above 12. Other metal ions are masked with triethanolamine.

4 QUALITY OF REAGENTS

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of test.

5 REAGENTS

5.1 Nitric Acid — 5 M (approx). Dilute 325 ml of nitric acid (r.d. 1.4) to 1 litre with water.

5.2 Hydrochloric Acid — 6 M (approx). Dilute 500 ml of hydrochloric acid (r.d. 1.19) to 1 litre with water.

5.3 Hydrochloric Acid — 1 M. Dilute 82 ml of hydrochloric acid (r.d. 1.19) to 1 litre with water.

5.4 Potassium Hydroxide Solution — 8 M (approx). Dissolve 449 g of potassium hydroxide per litre.

5.5 Triethanolamine Solution — Dissolve 100 g of triethanolamine [$(C_2H_5O_3)N$] in water and dilute to 1 litre.

5.6 Hydroxylamine Hydrochloride Solution — Dissolve 2 g of hydroxylamine hydrochloride ($NH_2OH.HCl$) in 100 ml of water.

5.7 Standard Calcium Solution — 0.01 M. Weigh 1 g of calcium carbonate, dried at a temperature not exceeding 200°C, to the nearest 0.001 g into a 500-ml beaker. Add 100 ml of water and add drop by drop from a burette the smallest (about 20 ml) of 1 M hydrochloric acid, necessary to dissolve the carbonate. Boil out the carbon dioxide, cool, and transfer quantitatively by rinsing with water into a 1 litre volumetric flask and dilute to the mark. Calculate the molarity of the standard calcium solution as follows:

$$M_{\mathrm{Ca}} = \frac{\text{mass of calcium carbonate, in g}}{100.1}$$

5.8 Solution of Disodium Salt of Ethylenediaminetetraacetic Acid (EDTA) — 0.01 M. Dissolve 3.80 g of the disodium salt of ethylene diaminetetraacetic acid ($C_{10}H_{14}O_8N_2Na_2.2H_2O$) in water in a 1 litre volumetric flask and dilute to the mark. Keep the solution in a polyethylene bottle, standardize the solution by adding to a conical flask 20.0 ml of 0.01 M standard calcium solution, 40 ml of water, 4 ml of potassium hydroxide solution, 2 ml of hydroxylamine hydrochloride solution and about 100 mg of cal-red indicator. Titrate also a blank solution containing all the reagents except the standard calcium solution, which is replaced by an equal volume of distilled water.

Calculate the molarity of the EDTA solution from the following equation:

$$M_{\mathrm{EDTA}} = \frac{a \times M_{\mathrm{Ca}}}{b - c}$$

where

a = volume of the standard calcium solution, expressed in ml;

b = volume of the EDTA solution, consumed by the standard calcium solution, expressed in ml;

c = volume of the EDTA solution, consumed in the blank determination, expressed in ml; and

M_{ca} = molarity of the standard calcium solution.

The solution mentioned under **5.4, 5.7** and **5.8** should preferably be stored in polyethylene bottles.

5.9 Cal-Red Indicator — Rub 100 mg of 2-hydroxy-1-(2-hydroxy-4-sulpho-1-naphthyl-azo)-3-naphthoic acid in a mortar with 10 g of sodium sulphate, to form a homogeneous mixture. Store in a dark bottle.

NOTE — When the sample contains more than 0.03 mg of copper the indicator is blocked. In this case use calcein as the indicator or mask the copper present by addition of approximately 5 ml of 1 percent solution of potassium cyanide.

6 APPARATUS

6.1 Dishes, of quartz, platinum or porcelain.

6.2 Weighing Balance, of suitable capacity having least count 0.01 mg.

7 PROCEDURE

7.1 Preparation of Test Sample

7.1.1 Weigh about 10 g of air dry pulp, previously torn to pieces of suitable size, to the nearest 0.01 g.

NOTE — For pulps containing more than 500 mg of calcium per kilogram, weigh an amount of pulp corresponding to a maximum of 5 mg of calcium or use a standardized 0.05 M EDTA solution for the titration.

7.1.2 At the same time weigh out a separate sample for the determination of dry matter content.

7.2 Determination

7.2.1 Ash the test sample in a dish as described in IS 6213 (Part 7).

NOTE — For cleaning of dishes, wash carefully; remove any spots in the dishes by rubbing with fine sand. Boil the dish four times with 6 M hydrochloric acid.

7.2.2 Rinse the inside of the dish with about 10 ml of water and add 3 ml of nitric acid. Heat the dish for 5 to 10 min on a steam-bath. If a brown precipitate of manganese dioxide is formed, filter the contents of the dish through a filter paper into a 300 ml conical flask and wash with water. If no insoluble residue is visible or if the residue is colourless, filtration is unnecessary. In that case rinse the contents of the dish directly into a 400 ml conical flask with the aid of water and adjust the volume to between 50 to 100 ml.

7.2.3 Add 5 ml of potassium hydroxide solution and add, after 5 min with occasional shaking of the flask, 5 ml of triethanolamine solution, 2 ml of hydroxylamine hydrochloride solution and about 100 mg of Cal-red indicator. Titrate with the EDTA solution to a colour change from wine-red to pure blue.

NOTE — The sum of calcium and magnesium can be determined at pH 10 by titration with 0.01 M EDTA solution using eriochrome black T as indicator. When more than 0.03 mg of copper is present the indicator is blocked. In this case, add approximately 5 ml of 1 percent solution of potassium cyanide.

7.3 Blank Test

To check that the new reagents do not contain any calcium, titrate a blank solution containing all the reagents and with the sample solution replaced by an equal volume of water.

8 CALCULATION

Carry out two determinations and calculate the results as follows:

$$\text{Calcium content, in mg/kg (parts per million)} = \frac{(d-e)\,M_{EDTA} \times 40.08 \times 1\,000}{m}$$

where

d = volume of the EDTA solution consumed by the sample, in ml;

e = volume of the EDTA solution consumed by the blank solution, in ml;

M_{EDTA} = molarity of the EDTA solution, in moles/L and

m = mass of pulp, calculated on an oven-dry basis, in g.

Report the results, in mg/kg (parts per million) as indicated below:

Mean of Duplicate Determinations mg/kg	*Nearest Unit of Report*
< 100	1
100 - 500	5
> 500	10

9 REPORT

The test report should state the results obtained and moreover, indicate all the conditions of the test, any details of procedure regarded as optional or not laid down in this standard and any incidents that may have affected the results.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Paper and Its Products Sectional Committee, CHD 15

Organization	*Representative(s)*
Central Pulp and Paper Research Institute, Saharanpur	Dr R. M. Mathur (***Chairman***)
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